

WO 2004/060804

PCT/AU2004/000005

3

starting materials, ie the salts, are completely dissolved as a first step. The process of Merck is preferably conducted at high temperatures between 900 and 1400°C and high pressures.

5

The present invention was developed with a view to providing an alternative process for the production of substantially discrete plate-like alpha alumina particles with a high aspect ratio.

10

It will be clearly understood that, although prior art use and publications are referred to herein, this reference does not constitute an admission that any of these form a part of the common general knowledge in the art, in Australia or in any other country.

15

Throughout this specification and in the appended claims, the term "comprising" is used inclusively, in the sense that there may be other features and/or steps included in the invention not expressly defined or comprehended in the features or steps subsequently defined or described. What such other features and/or steps may include will be apparent from the specification read as a whole.

20

25 Summary of the Invention

According to a first aspect of the present invention there is provided a process for the production of plate-like alumina particles with a high aspect ratio, the process comprising the steps of:

30

forming a mixture of nano-sized particles of an aluminium precursor compound and a sufficient volume fraction of a diluent; and

heat treating the mixture to form substantially discrete plate-like alpha alumina particles dispersed in the diluent.

35

It is to be clearly understood that the product of the process need not comprise only alpha alumina particles. For example, gamma alumina particles may also be present.

Moreover, the temperature for the step of heat treating may be selected to control the relative amounts of gamma alumina particles and plate-like alpha alumina particles that form. For example, if a preferred product requires
5 that only 50% of the particles be in the form of plate-like alpha alumina, the step of heat treating may be conducted at a lower temperature than that selected to produce a product requiring 90% plate-like alpha alumina particles.

10 Preferably the nano-sized particles of the aluminium precursor compound are substantially discrete.

Preferably, the process further includes the step of removing the diluent from the substantially discrete plate-
15 like alpha alumina particles after the step of heat treating.

The term "diluent" here is used to describe a substance in solid or liquid form that "dilutes" the mixture and is
20 added to help maintain separation of the particles of both the precursor alumina compound and/or the plate-like alpha alumina particles throughout the process. The diluent may react with the aluminium precursor compound or be present as a spectator.

25 Preferably, the diluent is soluble in a solvent which selectively removes the diluent and does not react with the plate-like alpha alumina particles and the step of removing the diluent after the step of heat treating comprises the
30 step of washing with the solvent. More preferably the solvent is water or an alcohol.

Preferably, the sufficient volume fraction of the diluent is at least 80% of the total volume of the mixture.

35 While a wide range of diluents may be used, the preferred diluents are selected to encourage plate-like growth of the alpha alumina particles during heat treatment. A preferred diluent is a metal salt such as sodium sulphate, potassium

sulphate or sodium chloride. Sodium chloride is highly preferred as being cheap and readily available.

5 A mineraliser in the form of a metal fluoride may also be added to the diluent to form a diluent-mineraliser system. The preferred metal fluorides are sodium fluoride, calcium fluoride, aluminium fluoride and sodium aluminium fluoride (cryolite).

10 The conditions for heat treatment of the mixture depend on the particular diluent or diluent-mineraliser system used. The advantage of using a diluent-mineraliser system is that the step of heat treating may be conducted at a lower temperature than for a diluent used alone.

15 Preferably, the step of heat treating the mixture is conducted below the melting point of the diluent or below the liquidus of the diluent-mineraliser system in order to maintain separation between the plate-like particles as
20 they form. Alternately, the step of heat treating the mixture is conducted above the melting point of the diluent or above the liquidus of the diluent-mineraliser system. When this alternative is used, the process further comprises the step of stirring the mixture during the step
25 of heat treating to minimise interlocking of the growing plate-like alpha alumina particles.

Any suitable process for stirring of the mixture may be employed including, but not limited to, mechanical mixing,
30 rotation of the container, the use of convection currents, induction heating or any other method that imparts relative motion of the particles in the mixture.

Preferably, the aluminium precursor compound is aluminium
35 hydroxide. Aluminium hydroxide is cheap, readily available, easy to handle and readily dehydrates to form aluminium oxide.

The process described in WO 99/59754 and incorporated

herein by reference may be used to produce substantially discrete nano-sized particles of an alumina precursor compound as the starting material for the process according to the first aspect of the present invention.

5

Other suitable aluminium precursor compounds include aluminium sulphate, aluminium nitrate and aluminium chloride. With these precursor compounds, the process preferably further comprises the steps of milling the aluminium precursor compound with a suitable diluent such as sodium hydroxide in accordance with the method described in the applicant's International application WO 99/59754, the contents of which are incorporated herein by reference.

15 According to a second aspect of the present invention there is provided a process for the production of substantially discrete ultrafine plate-like alumina particles having a high aspect ratio, the process comprising:

20 milling a mixture of an aluminium precursor compound and a sufficient volume fraction of a diluent to form a dispersion of nano-sized particles of an intermediate aluminium compound in the diluent; and,

thereafter heat treating the dispersion to convert the nano-sized particles of the intermediate aluminium compound to substantially discrete plate-like particles of alpha alumina.

Preferably the process further comprises the step of removing the diluent such that ultra-fine plate-like particles are left behind in the form of an ultrafine powder. Preferably the step of removing the diluent includes the step of washing with a solvent which selectively dissolves the diluent while not reacting with the plate-like alumina particles.

35

The intermediate aluminium compound would typically be aluminium hydroxide or aluminium oxide. The precursor aluminium compound would typically be aluminium hydroxide.

According to a third aspect of the present invention there is provided a product in accordance with the processes described above. Such a product is suitable for use in the following applications: soft focus cosmetics, pearlescent pigments, ceramic components, die coatings, and hard coatings.

According to a fourth aspect of the present invention there is provided plate-like alpha alumina particles having an aspect ratio of width to diameter of between 1:10 and 1:100 and more preferably between 1:20 and 1:50. The preferred aspect ratio depends on the particular application for which the powders are required. Preferably the plate-like alumina particles have a diameter between 0.1 to 30 microns. Preferably the plate-like alumina particles have a thickness of between 50 and 200 nm.

Brief Description of the Drawings

In order to facilitate a more detailed understanding of the nature of the invention, preferred embodiments of the process for the production of ultrafine plate-like alumina particles will now be described in detail, by way of example, only, with reference to the accompanying drawings, in which:

Figure 1 is a Scanning Electron Micrograph of alumina plate-like particles in accordance with Example 1;

Figure 2 is a Scanning Electron Micrograph of alumina plate-like particles, many of which are interlocked due to intergrowth made in accordance with Example 2;

Figure 3 is a Scanning Electron Micrograph of alumina plate-like particles made in accordance with Example 3;

Figure 4 is a Scanning Electron Micrograph of alumina plate-like particles made in accordance with Example 4;

Figure 5 is a graphical representation showing the size distribution of the diameter of the plate-like particles of Figure 4;

Figure 6 is a Scanning Electron Micrograph of alumina plate-like particles made in accordance with Example 5;

CLAIMS:

1. A process for the production of plate-like alumina particles comprising the steps of:

forming a mixture of nano-sized particles of an aluminium precursor compound and a sufficient volume fraction of a diluent; and

heat treating the mixture to form substantially discrete plate-like alpha alumina particles dispersed in the diluent.

2. A process for the production of plate-like alumina particles according to claim 1 further comprising the step of removing the diluent after the step of heat treating.

3. A process for the production of plate-like alumina particles according to claim 2 wherein the diluent is soluble in a solvent and the step of removing the diluent from the mixture comprises the step of washing with the solvent after the step of heat treating.

4. A process for the production of plate-like alumina particles according to claim 3 wherein the solvent is water or an alcohol.

5. A process for the production of plate-like alumina particles according to any one of the preceding claims wherein the sufficient volume fraction of the diluent is at least 80% of the total volume of the mixture.

6. A process for the production of plate-like alumina particles according to any one of the preceding claims wherein the diluent is a metal salt.

7. A process for the production of plate-like alumina particles according to claim 6 wherein the metal salt is selected from the group comprising; sodium sulphate, potassium sulphate and/or sodium chloride.

8. A process for the production of plate-like alumina particles according to any one of the preceding claims further comprising the step of adding a mineraliser to the diluent to form a diluent-mineraliser system.

9. A process for the production of plate-like alumina particles according to claim 8 wherein the mineraliser is a metal fluoride.

10. A process for the production of plate-like alumina particles according to claim 8 wherein the metal fluoride is selected from the group comprising further comprising; sodium fluoride, calcium fluoride, aluminium fluoride and/or sodium aluminium fluoride.

11. A process for the production of plate-like alumina particles according to any one of claims 1 to 7 wherein the step of heat treating the mixture is conducted below the melting point of the diluent

12. A process for the production of plate-like alumina particles according to any one of claims 8 to 10 wherein the step of heat treating the mixture is conducted below the liquidus of the diluent-mineraliser system.

13. A process for the production of plate-like alumina particles with a high aspect ratio according to any one of claims 1 to 7 wherein the step of heat treating the mixture is conducted above the melting point of the diluent.

14. A process for the production of plate-like alumina particles with a high aspect ratio according to any one of claims 8 to 10 wherein the step of heat treating the mixture is conducted above the liquidus of the diluent-mineraliser system.

15. A process for the production of plate-like alumina particles with a high aspect ratio according to claim 14 further comprising the step of stirring the mixture during

the step of heat treating.

16. A process for the production of plate-like alumina particles according to any one of the preceding claims wherein the aluminium precursor compound is selected from the group comprising; aluminium hydroxide, aluminium sulphate, aluminium nitrate and/or aluminium chloride.

17. A process for the production of plate-like alumina particles according to any one of preceding claims wherein the nano-sized particles of the alumina precursor compound are substantially discrete.

18. A process for the production of ultrafine plate-like alumina particles having a high aspect ratio comprising the steps of:

milling a mixture of an aluminium precursor compound and a sufficient volume fraction of a diluent to form a dispersion of nano-sized particles of an intermediate aluminium compound in the diluent; and,

thereafter heat treating the dispersion to convert the nano-sized particles of the intermediate aluminium compound to substantially discrete plate-like particles of alpha alumina.

19. A process for the production of plate-like alumina particles according to claim 18 further comprising the step of removing the diluent from the mixture after the step of heat treating.

20. A process for the production of plate-like alumina particles according to claim 19 wherein the step of removing the diluent comprises the step of washing with a solvent which selectively dissolves the diluent while not reacting with the plate-like alumina particles.

21. A process for the production of plate-like alumina particles according to any one of claims 18 to 20 wherein the precursor aluminium compound is aluminium hydroxide or

aluminium oxide.

22. Plate-like alpha alumina particles produced by the process according to any one of the preceding claims.

23. Plate-like alpha alumina particles having an aspect ratio of width to diameter of between 1:10 and 1:100 and a diameter between 0.1 to 30 microns.

24. Plate-like alpha alumina particles having an aspect ratio of width to diameter of between 1:20 and 1:50 and a diameter between 0.1 to 30 microns.

25. Plate-like alpha alumina particles according to claim 23 or 24 having a thickness of between 50 and 200 nm.

26. Plate-like alpha alumina particles produced as herein described with reference to and as illustrated in any one of the Examples 1 to 5.

27. A process for the production of plate-like alumina particles as herein described with reference to and as illustrated in any one of the Figures 1 to 7.